APPLICATION NOTE

Infrared Spectroscopy

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Industrial Equipment Failure Analysis Using FT-IR Microscopic Techniques

Introduction

Fourier Transform Infrared (FT-IR) Spectroscopy is used routinely in the analysis of both new and in-service lubricants to determine chemical attributes such as oxidation, nitration, sulfation, water and soot.[1] These chemical species are normally attributed to the chemical stress and oxidation of the fluid and can be monitored using trending or differential techniques.[2] FT-IR analysis is also used routinely to monitor depletion of lubricant additives such as detergents and corrosion inhibitors. In addition to in-service monitoring, FT-IR spectroscopy has proven to be a valuable tool for identification of contamination in lubricants and for analyzing chemical species found on failed components (varnishes/sludges).[3] In such analyses, the qualitative and quantitative information of the bulk solution/sample is tested while little attention is given to the micro-environment except for particle counts. Traditionally, the analysis of particles in small lubricant and grease samples has been problematic especially when trying to determine failure modes of lubricants for root cause analyses.

A valuable accessory to the FT-IR instrument that can be used for the analysis of micro-environments in lubricants and grease is the FT-IR microscope. The first commercially available FT-IR microscope was invented by PerkinElmer and Vincent Coates in 1953.[4] Continuous improvements in system hardware combined with intelligent software development has led to highly automated modern-day systems.

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Today, FT-IR microscopes can be fitted with an automated x, y, z stage that has micron level accuracy allowing for precise movements. These systems are routinely used in the pharmaceutical, plastics and forensics industries to analyze small and difficult samples in-situ. In practice, a sample is presented to the FT-IR microscope by placement on the automated stage. The visible image of the sample area is collected for reference and visual analysis. The user can set the position of any region of interest of the visible image using a number of IR techniques which are suggested and described in ASTM E334 - Standard Practice for General Techniques of Infrared Microanalysis.[5]

The main advantage of the FT-IR microscope is the ability to focus the IR energy on sample sizes ranging from 5 to 500 µm in size using optical apertures. Once the sample position is identified, the FT-IR microscope may be operated in transmission, reflectance or ATR modes allowing for the easy analysis of liquids, solids, viscous and difficult sample types. The reflectance and ATR modes of analysis have recently been used to effectively analyze lubricant deposits in-situ on the surface of gears, bearings, filters and other industrial components.[6] Regions of interest as single location markers, line scans, map scans and imaging scan options all may be used to collect the IR spectral data of the sample.

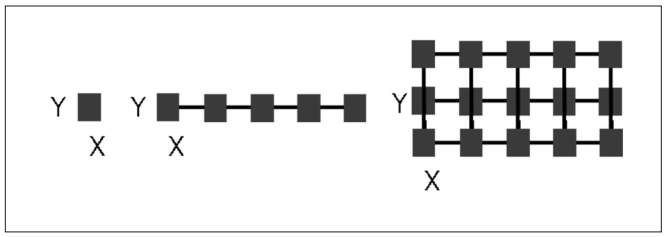

Figure 1. Single marker, line scan and map scan settings.

Lubrication Failure Analysis of a Kingsbury® Bearing

Kingsbury bearings surround a shaft with several interlocking shoes which float on a hydrodynamic layer of lubricant flowing across the bearing surfaces. Figure 2 shows a clean bearing shoe compared to the failed shoe (with visible varnish deposition and wear present). To gain further insight as to how the deposits formed, FT-IR microscopy analysis was carried out on the surface species present on the sample.

The bearing shoe was placed on a slide holder and affixed to the microscope stage. The stage was centered and a visible image of the sample was obtained. Using the visible image of the sample, a 10 x 10 point map containing 100 individual spectra was defined. The spectra were collected using a 100 x 100 µm aperture size with 100 µm spacing between the markers.

Once the map scan has been collected, an FT-IR spectrum can be viewed from any point on the map in order to show the change of chemistry across different sections. Figure 3 shows a spectral overlay from three different points. Analysis of the resulting spectra reveal hydroxyl group, thermal-decomposition and oxidation increases of the fluid as well as deposition of the phosphate anti-wear additive.

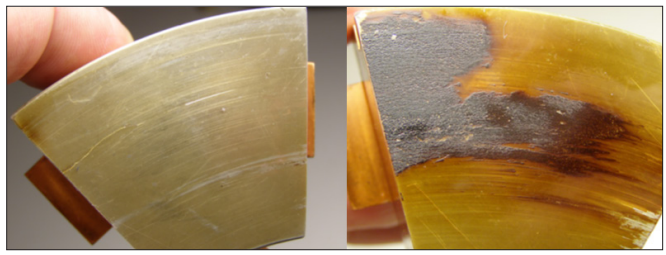

Figure 2. Clean Kingsbury bearing shoe compared to failed shoe.

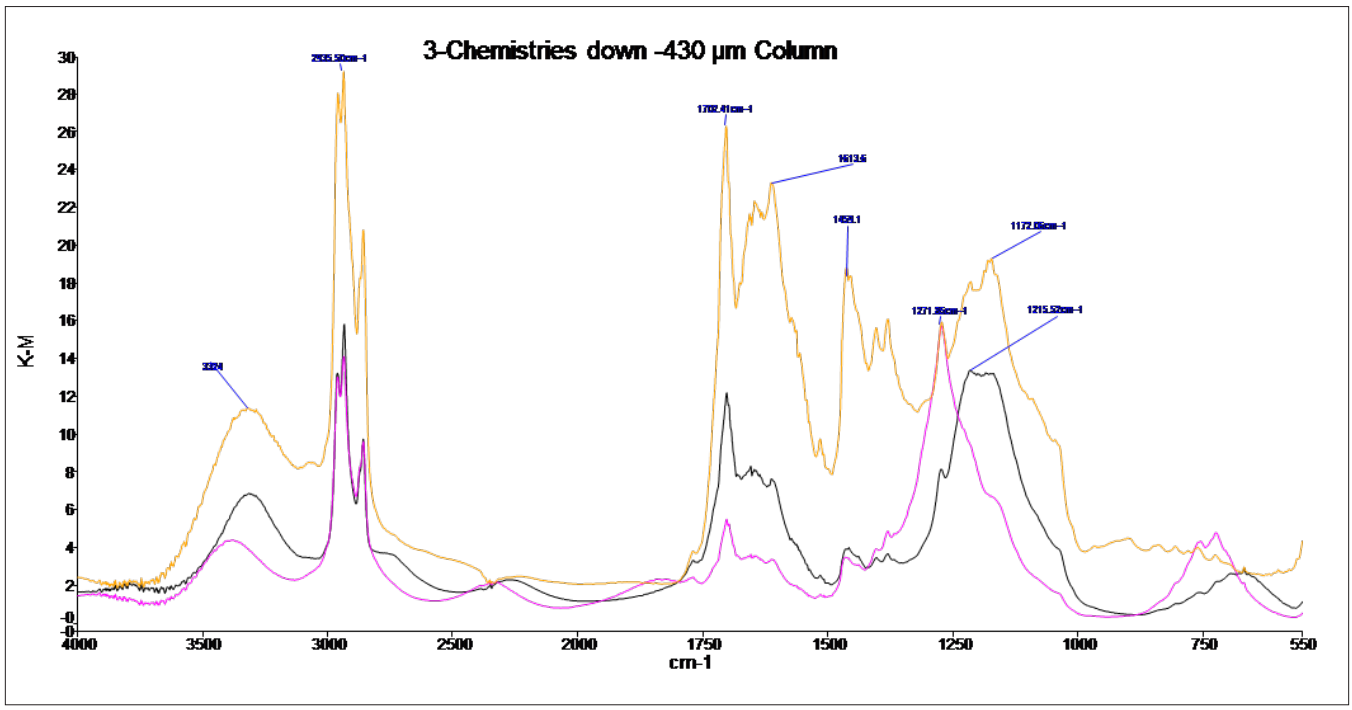

Figure 3. Spectral overlay of three different points on map scan of Kingsbury bearing.

Furthermore, total absorbance plots at individual wavenumber values can be prepared to see high and low concentrations of individual chemical species on the surface of the sample. Figure 4 illustrates four color images of the surface at different wavenumbers showing how the chemistries are not evenly distributed across the surface.

In this example we showed that FT-IR microscopy analysis was able to elucidate the cause of the surface species on the Kingsbury bearing to be determined, reflected by the non-homogeneity in surface chemistry. Consequently, understanding these surface deposits allows actionable measures to be implemented to prevent this recurrence on other equipment.

FT-IR Microanalysis of In-Line Actuator Filters

It is common for filters to fail over time due to the collection of debris on the surface. This can cause them to plug which often results in pressure problems throughout the system. Replacing the filter is a simple way to resolve the current issue but determining the initial cause of the debris can help to mitigate future problems.

An in-line last chance filter is usually placed before an Electrohydraulic Control (EHC) valve to prevent actuator seizing and provide protection from contamination. These filters have pore sizes of approximately 150 µm and can become plugged with various materials.

For other types of analysis, the filter contaminants would have to be removed individually with tweezers. However, FT-IR microscopy analysis simply requires the filter to be mounted on a slide and placed on the microscope stage. For this filter, a single marker analysis was carried out, using a 162 x 26 µm aperture size along the length of the fiber, as shown in Figure 5.

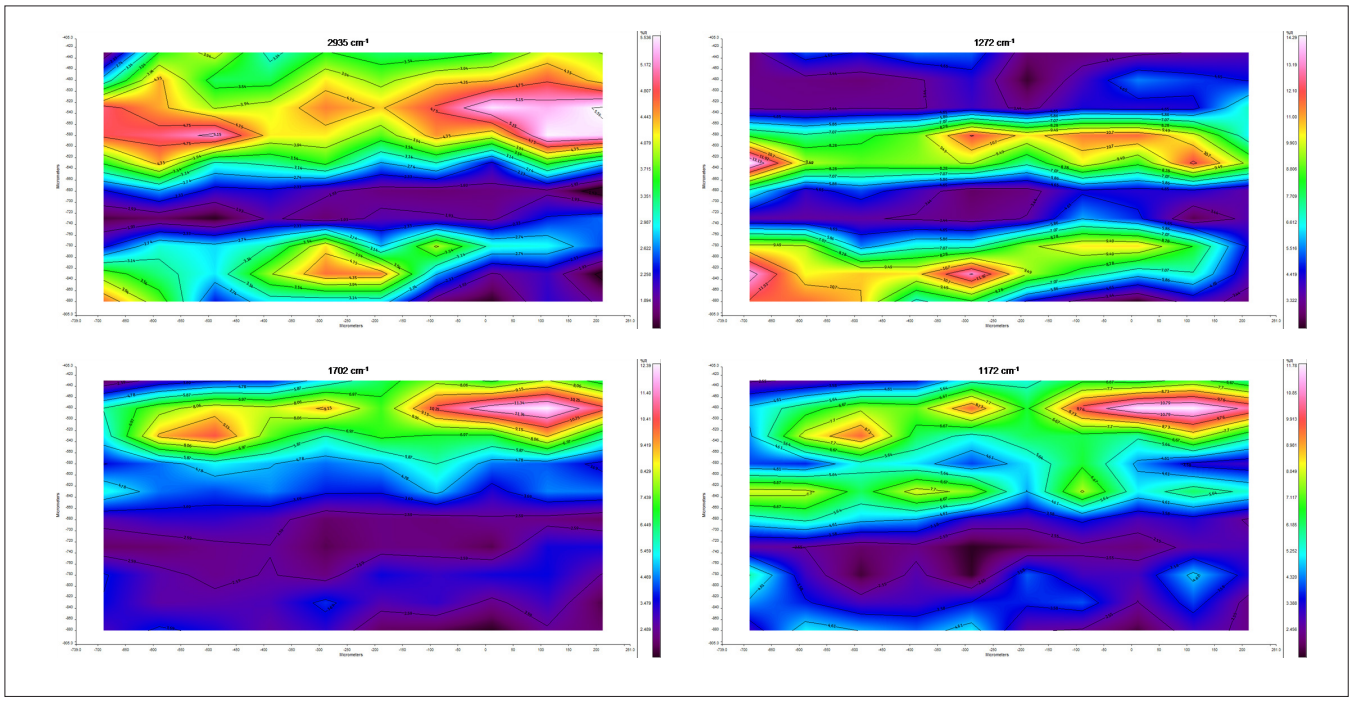

Figure 4. False color images of surface of Kingsbury bearing at different wavenumbers.

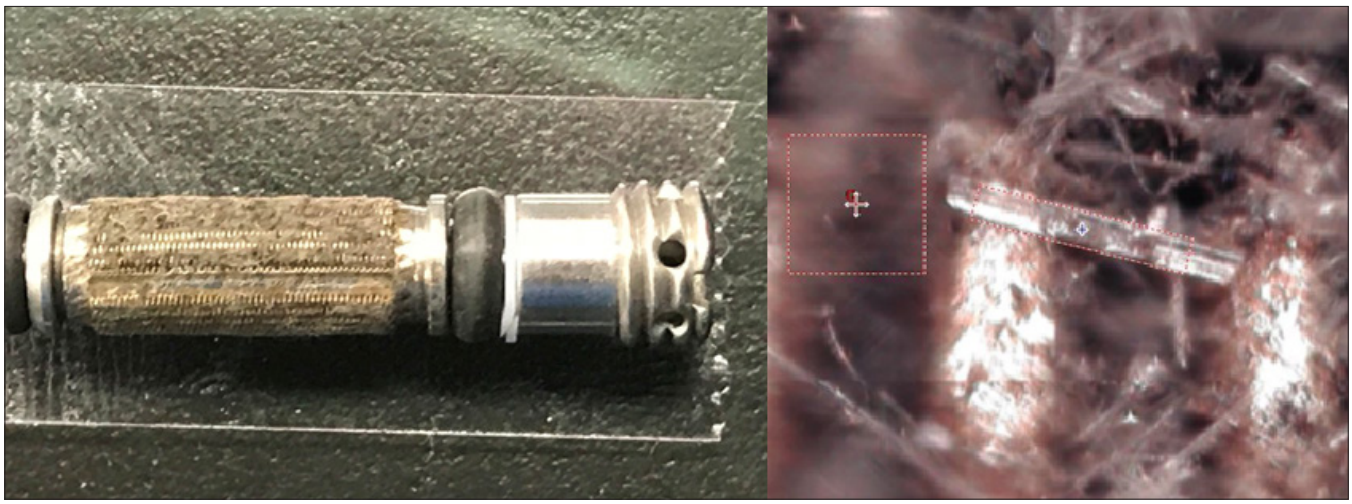

Figure 5. Failed in-line actuator filter on microscope plate (left) and collected visible image (right).

The resultant spectrum was identified as fiberglass. There are other filters in the system made from fiberglass material and, as such, it can be proposed there is a problem upstream with another filter.

In a separate case study, a system was having actuator plugging with poor fluid flow and it was believed to be due to a broken filter. Similar to the previous example, FT-IR microscopy was used to analyze individual fibers on the surface of the filter. The collected spectra showed the unexpected presence of C≡N (nitrile) and NH_2 (amine) bands. Further investigative work was carried out by removing the contaminating fibers from the filter and analyzed using a map scan, as shown in Figure 6.

Spectra from different points of the map scan were analyzed and it was determined that some of the contaminating fibers were hair. Furthermore, some of the mass containing CN vibrations was found to be from acrylonitrile o-rings.

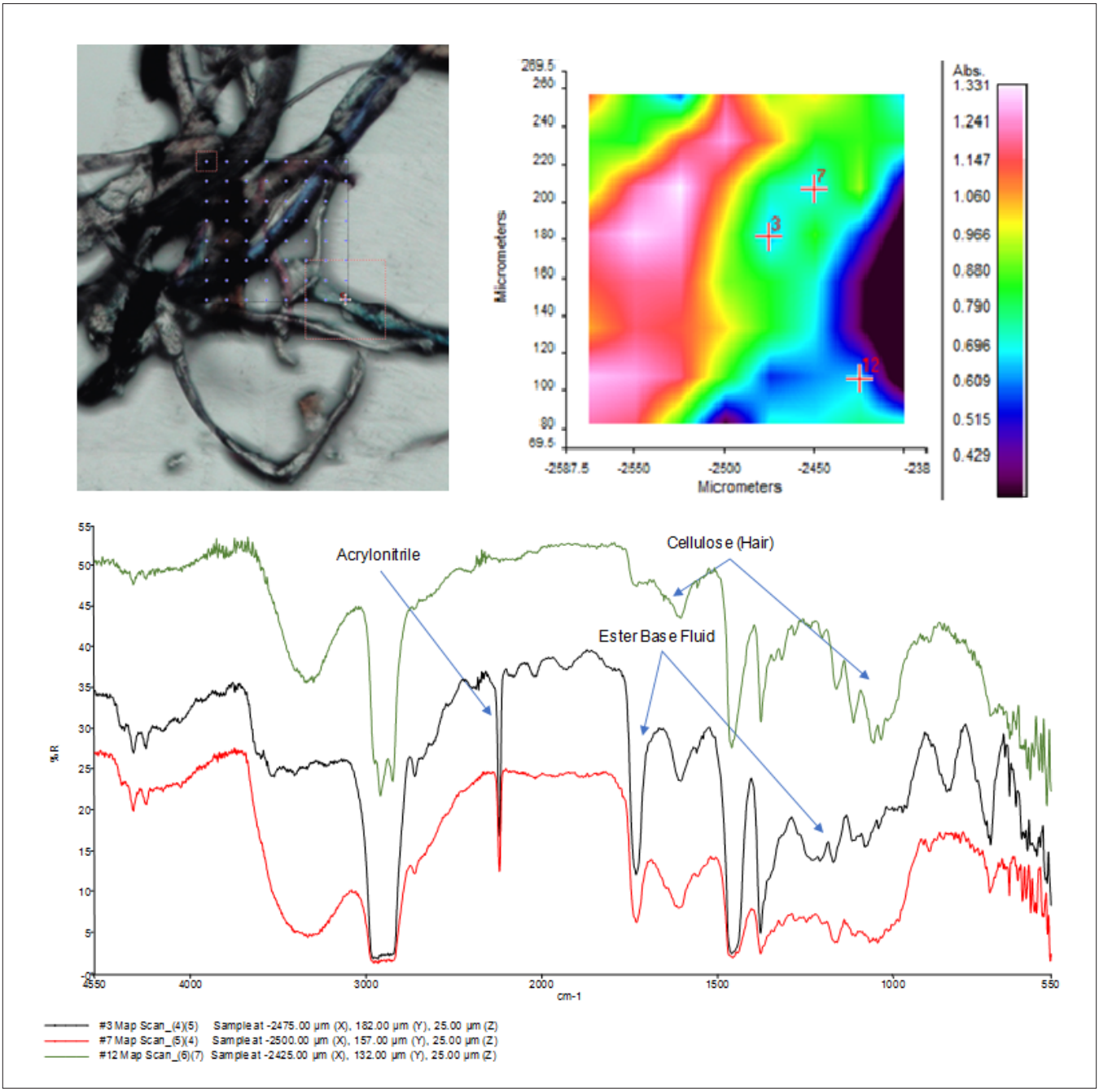

Figure 6. Visible image of extracted fibers (top left), color image of map scan (top right) and spectra from three points of map scan (bottom).

Hydraulic Control Valve Failure Analysis

The hydraulic control valve, shown in Figure 7, malfunctioned due to the observed corrosion of the brass surface resulting in fluid leakage. FT-IR microscopy can be used to determine how the pits and deposits on the surface of the valve were formed.

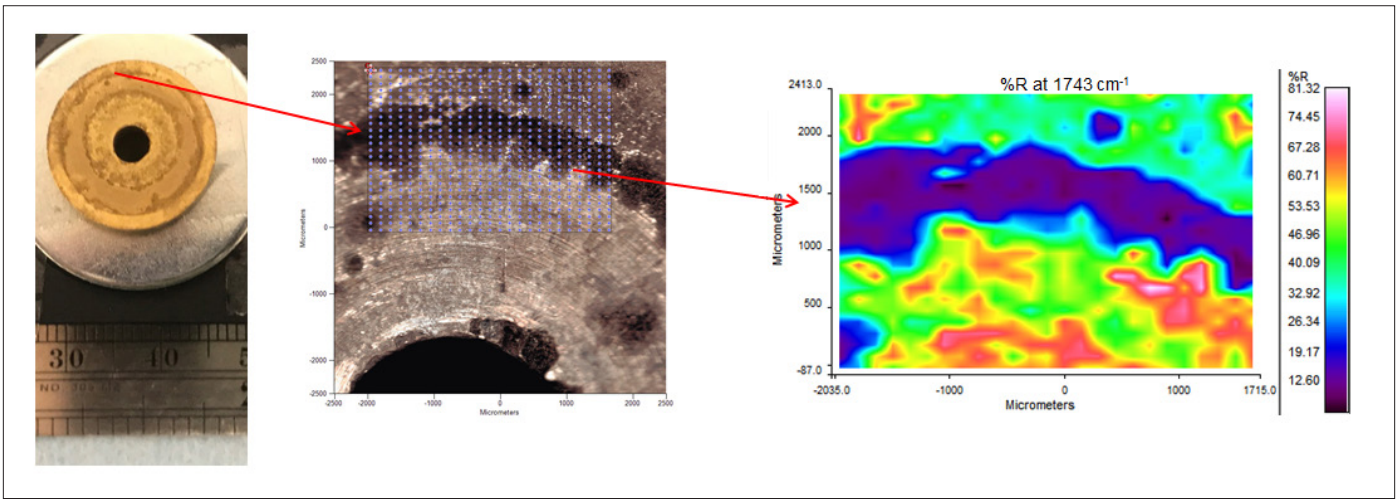

Figure 7. Failed hydraulic control valve (left), collected visible image (middle) and color image at 1743 cm^{-1} (right).

A 5000 x 5000 µm visible image was taken and a large 25 x 25 point map scan, totaling 625 spectra, was collected from a region of this visible image. The map was plotted as a 3D color image using the 1743 cm^{-1} ester bands of the spectra.

Studying the spectra within the corrosion pits allowed one to determine that this system was using a vegetable oil based hydraulic fluid that was inappropriate for the specific application. Heat, water and copper from the brass valve caused oxidation reactions to occur resulting in the formation of acids. The acids then reacted with the surface of the valve, causing it to corrode; leaving the resultant acid salts in the reacted holes. Determining the cause of the valve surface corrosion allowed the system reliability and efficiency to be improved by changing to a different hydraulic fluid.

Conclusion

These case studies demonstrate that FT-IR microscopy analysis is capable of determining the cause of failures of many different pieces of industrial equipment. This analysis can then allow the user to take preventative action measures in order to improve the reliability of the system and minimize future failures.

References

1. Annual Book of ASTM Standards (2010) - D7669 Standard Guide for Practical Lubricant Condition Data Trend Analysis
2. Annual Book of ASTM Standards (2010) - E2412 Standard Practice for Condition Monitoring of Used Lubricants by Trend Analysis Using Fourier Transform Infrared (FT-IR) Spectrometry
3. J.Fitch, Sludge and Varnish in Turbine Systems, Machinery and Lubrication Magazine
4. 60 Years of PerkinElmer Innovation in Infrared Spectroscopy (2005), PerkinElmer
5. Annual Book of ASTM Standards (2010) - E334 Standard Practice for General Techniques of Infrared Microanalysis
6. Aditya, M., Amarnath, P.K. Kankar, Failure Analysis of a Grease-Lubricated Cylindrical Roller Bearing, Procedia Technology, 14 (2014): pg 59-66.

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